

SECOND QUARTERLY REPORT

CHARACTERIZATION OF RECOMBINATION AND CONTROL ELECTRODES FOR SPACECRAFT NICKEL-CADMIUM CELLS

by

W. N. Carson, Jr., G. Rampel and I. B. Weinstock

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1.0 INTRODUCTION AND SUMMARY

1.1 Task I - Oxygen Sensing Electrode

The objective of this task is to develop an oxygen sensing electrode that will give a linear (or a reasonably close to linear) response to oxygen partial pressure in the range of approximately 5 to 30 PSIA. The initial approach being investigated is the use of diffusion controlled electrode reaction rates.

During the second quarter the following phases of this task were accomplished:

1. A complete set of polarization studies were made on eleven dual test electrodes at 0, 5, 10, 15, 20, 25, and 30 PSIA oxygen pressure at room temperature.
2. The more promising electrodes were evaluated over the temperature range of -10°C to 35°C .

1.2 Task II - Oxygen Recombination Electrode

The objective of this task is to select an oxygen recombination electrode capable of recombining the oxygen generated by the positive plate during charging. This electrode must be able to function over the temperature range of -20°C to $+40^{\circ}\text{C}$. Selection of electrodes will be based upon their polarization characteristics.

During the second quarter the following phases of this task were accomplished:

1. Test cells for the characterization of electrodes were modified to improve heat transfer between the working electrodes and the atmosphere.

2. The ability of our electrode fabrication process to produce electrodes of consistent quality has been demonstrated.
3. The effect of "ageing" on the performance of electrodes has been investigated, and no significant effects have been observed.
4. The performance of a number of fuel cell electrodes have been investigated. Electrodes developed in this program offer the best performance.

1.3 Task III - Negative Plate Evaluation

The objective of this task is to select suitable lots of negative plate for use in prototype and final cell assembly. Lots will be selected upon their recombination ability and ampere-hour stability.

During the second quarter three lots of negative plate were received from manufacturing and evaluated. The lot with the best balance of properties has been retained for use in the construction of prototype and final cells.

1.4 Task IV - Assembly of Plate Packs with Recombination Electrodes

The objective of this task is to apply the results of Task II and assemble plate packs with recombination electrodes for use in prototype cells. During this quarter the plate was formed, and the packs, recombination electrodes, and separator were sent to the Research and Development Center to be used in assembly of prototype cells.

2.0 TECHNICAL DISCUSSION

2.1 Task I - Oxygen Sensing Electrode

2.1.1 Experimental Details

The equipment and test cells were described in detail in the first quarterly report and have not been changed. In the tests it was not necessary to use the oxygen pressure regulating system, as the polarization data could be taken over a short period of time during which the oxygen pressure in the test cell remains constant. The pressure regulating equipment will be used in long term studies and response tests.

The polarization test consists of measuring the current and voltage of the sensing electrode connected through a known load resistance to a charged cadmium electrode. A polarization curve is run for each oxygen pressure. The performance of the electrode at constant oxygen pressure is of importance only with respect to the stability of the electrode response under a fixed load. Data collected under constant load versus oxygen pressure is needed to obtain the signal response of the electrode.

2.1.2 Results and Discussion

Details of the electrodes evaluated are presented in Table I. Large (9.8 cm^2 area) and small (0.85 cm^2 area) electrodes of all eleven materials were tested.

Typical polarization curves are shown in Figure 1. The curves are for the large electrode made of a porous nickel substrate covered with a one mil thick Teflon film. Typical response curves are presented in Figures 2

through 6. All data are for 25°C.

The desired response is an electrode signal that is linearly related to the oxygen pressure in the cell. Some types of non-linear response are potentially almost as useful, however. The response shown in Figure 2 is not useful as a signal generating electrode as the slopes of the curves are too shallow and they flatten out at higher gas pressures. The responses shown in Figures 3, 4, 5, and 6 are useful, for the most part, even if they are not linear.

An analysis of the plots of the signal response vs. oxygen pressure for all eleven electrode materials, at 25°C, is presented in Table II. The results indicate that all of the substrate materials are potentially useful as signal electrodes and that the film thickness is critical to electrode performance. The results also give some indication that the test cell construction may also be critical. Some of the failures listed in Table II may be due to the cell construction, although, as far as is known the construction of all cells is very similar.

Polarization runs of nickel mesh, nickel sinter, and gold mesh electrodes were performed at various temperatures. Results are presented in Table III. The influence of temperature is shown graphically in Figures 7 and 8, in which the signal response is plotted as a function of temperature at a fixed pressure.

TABLE I
SIGNAL ELECTRODE MATERIALS

NO.	SUBSTRATE MATERIAL	FILM THICKNESS (1,2)	SUBSTRATE DESCRIPTION
1	Nickel Sinter	1 mil	0.020" porous nickel (nominal) 4-7 micron pores, 80% total porosity. Support: Exmet 5 Ni 10-1/0. General Electric, Gainesville, Florida
2	Nickel Sinter	0.5 mil	DO
3	Nickel Sinter	0.25 mil	DO
4	Nickel Mesh	1 mil	Exmet nickel mesh 5 Ni 10-3/0. Exmet Corporation, Tuckahoe, N.Y.
5	Nickel Mesh	0.5 mil	DO
6	Nickel Mesh	0.25 mil	DO
7	Gold Mesh	1 mil	100 X 100 screen 0.003" pure gold wire. Cold Roscoe Mfg. Co., So. Norwalk, Connecticut
8	Gold Mesh	0.5 mil	DO
9	Gold Mesh	0.25 mil	DO
10	Platinum-coated Tantalum Mesh(3)	1 mil	0.010" expanded tantalum mesh one face covered with 0.00025" bright platinum. Anode stock, Metal and Controls, Inc., Attleboro, Mass.
11	Platinum Coated Tantalum Mesh (4)	1 mil	0.0010" expanded tantalum mesh, two faces with 0.00025" bright platinum Anode stock, Metal and Controls, Inc., Attleboro, Mass.

NOTES TO TABLE I

1. Films were Teflon skived films, Dilectrix Corporation, Farmingdale, N.Y.
2. Films were applied as follows:

1 mil	12,000 PSI at 672°F for ca. 45 min.
0.5 mil	8,000 PSI at 672°F for ca. 60 min.
0.25 mil	6,000 - 8,000 PSI at 672°F for ca. 60 min.

 All electrodes are air cooled under pressure to room temperature
3. Mesh approximates Exmet 3/10 style. No designation given by manufacturer.
4. Mesh approximates Exmet 3/10 style, but is ribbed so that adjacent strands are not in same plane.

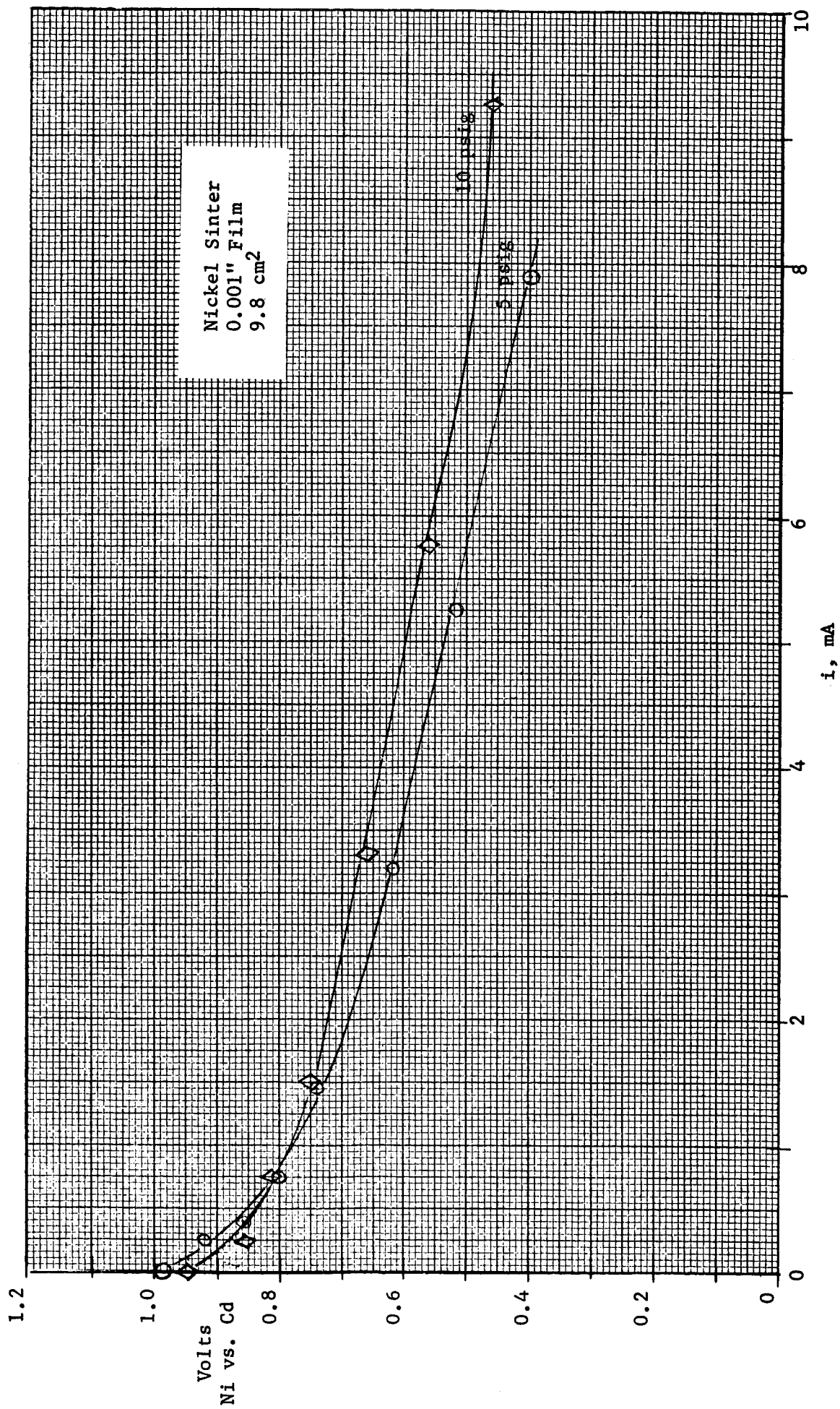


Figure 1: Typical Polarization Curve

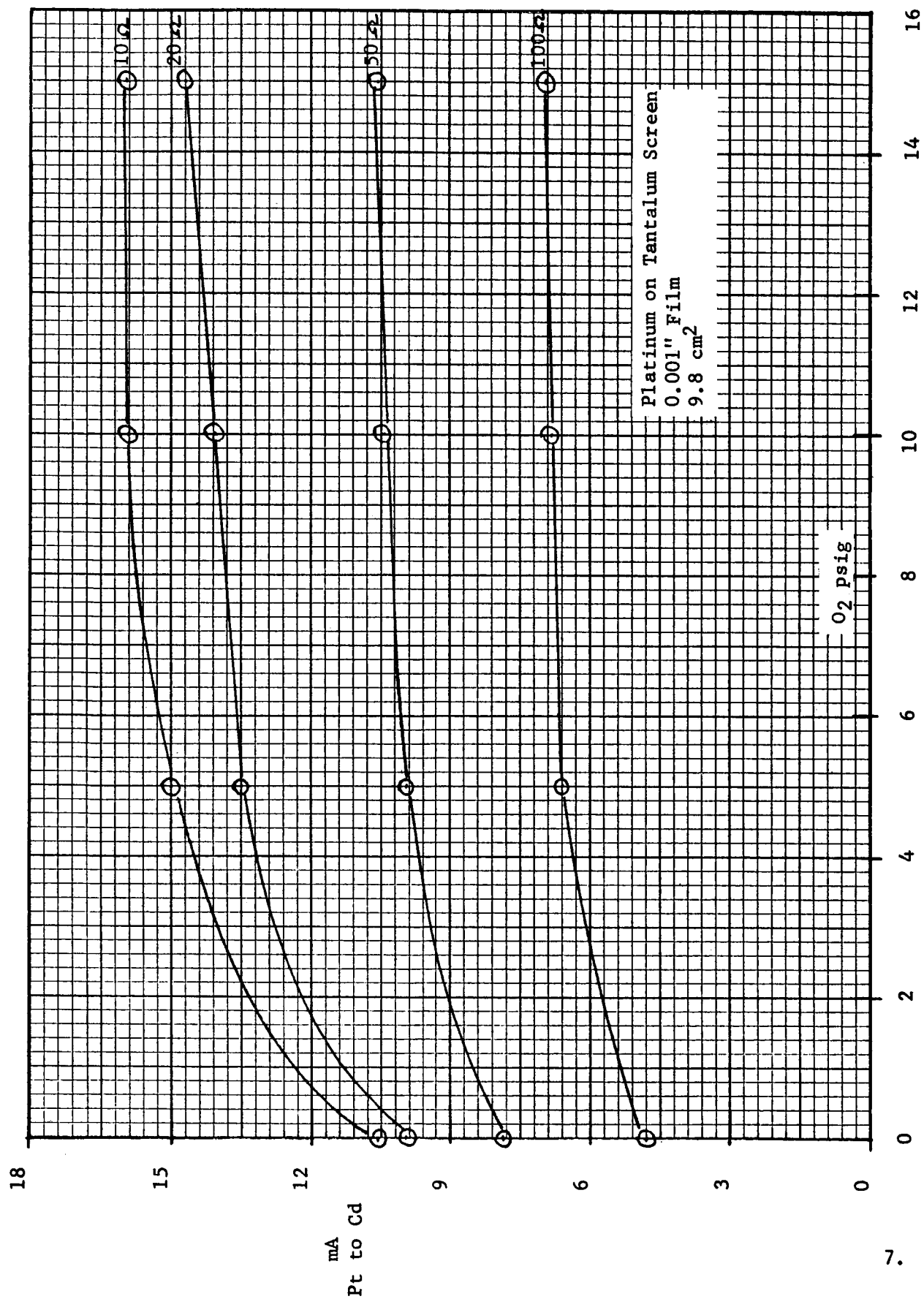


Figure 2: Electrode Response - I

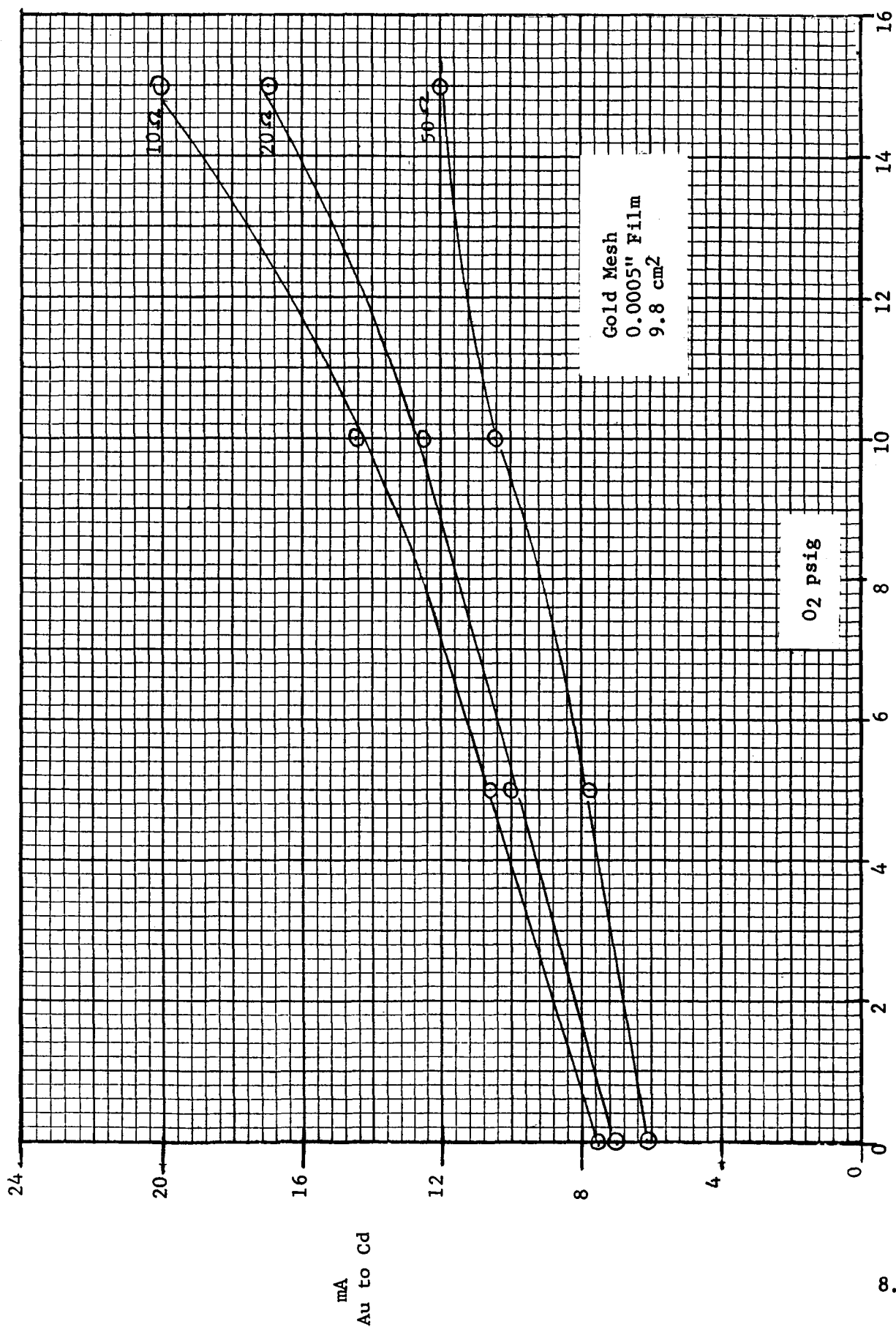


Figure 3: Electrode Response - II

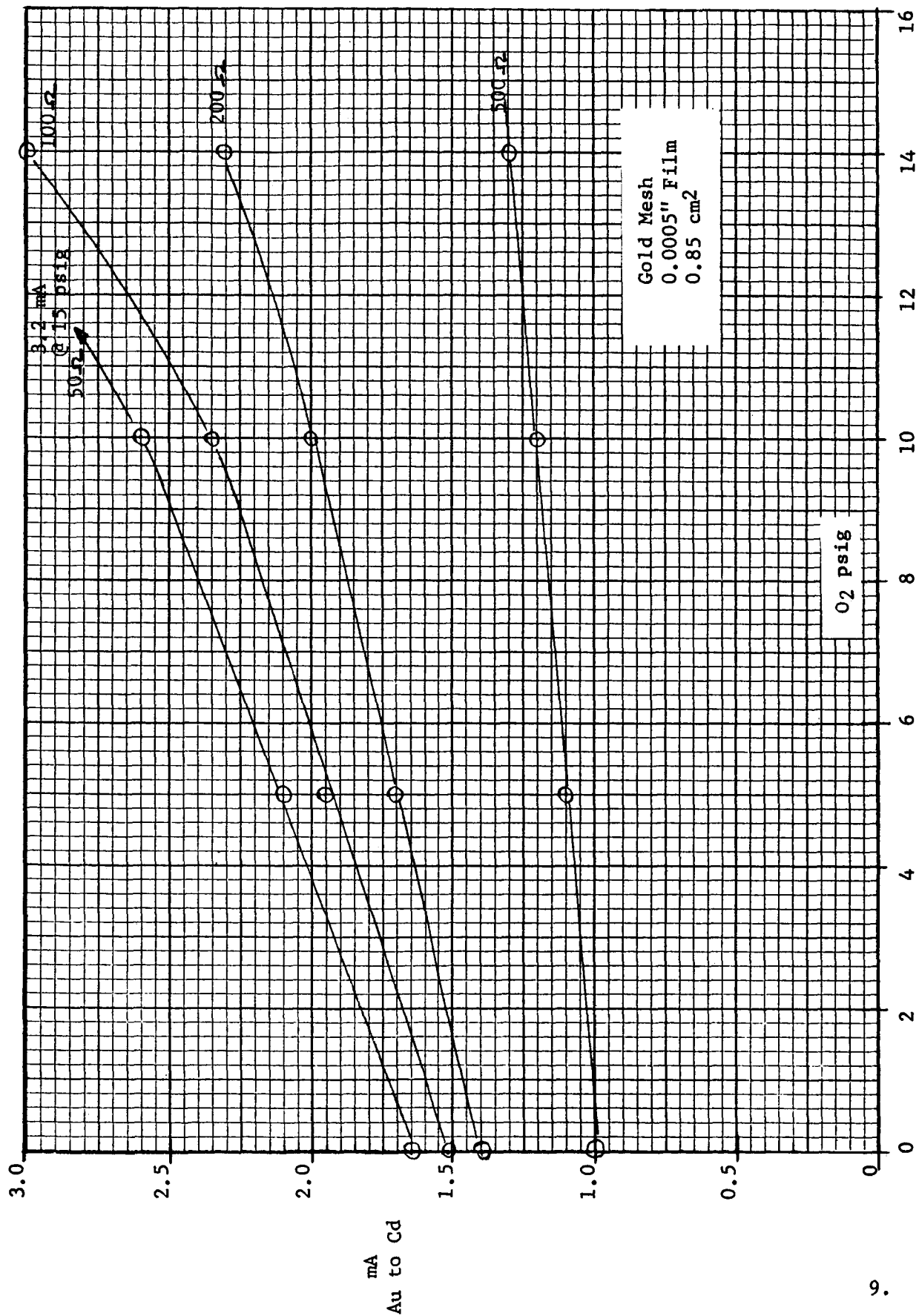


Figure 4: Electrode Response - III

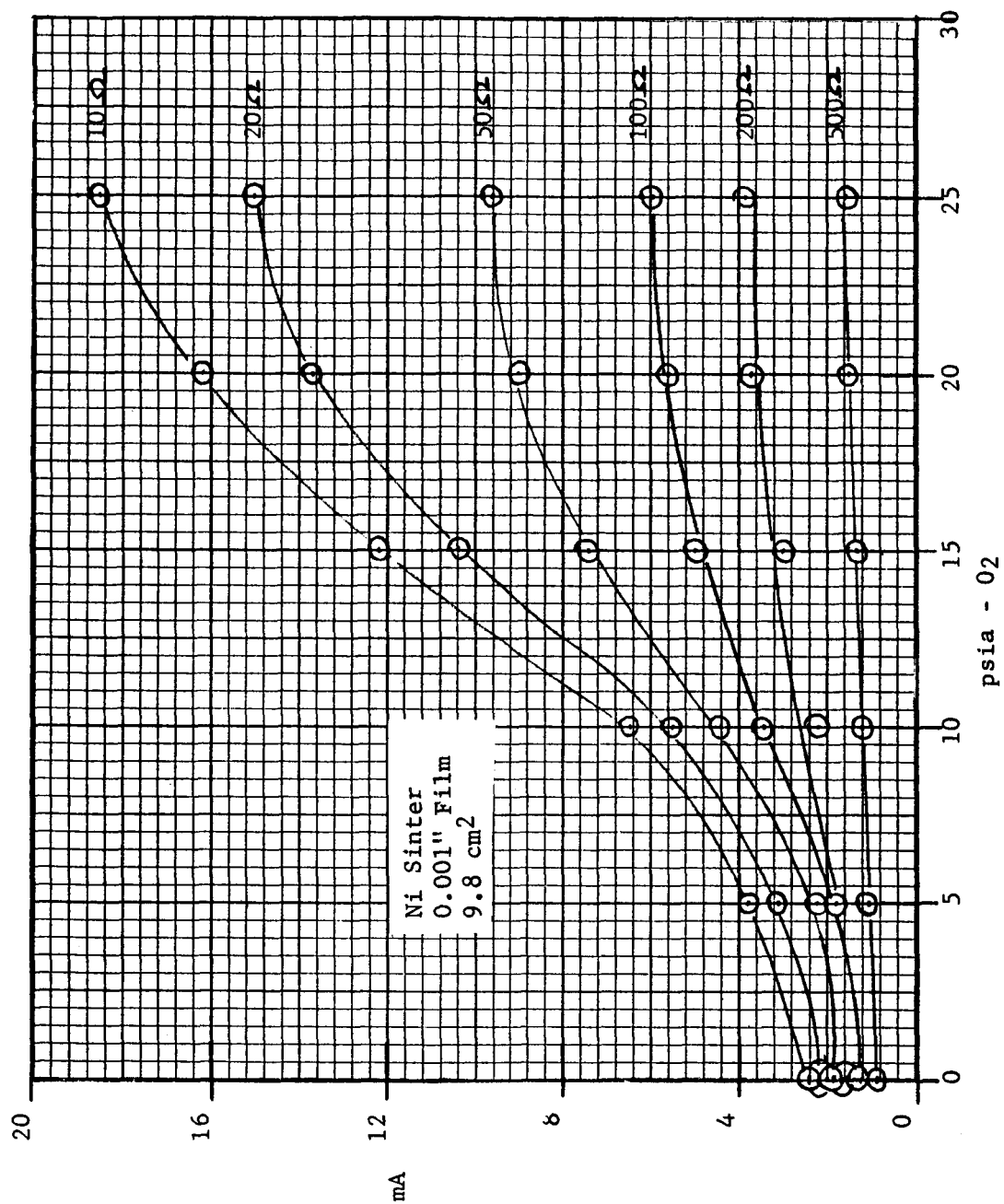


Figure 5: Electrode Response - IV

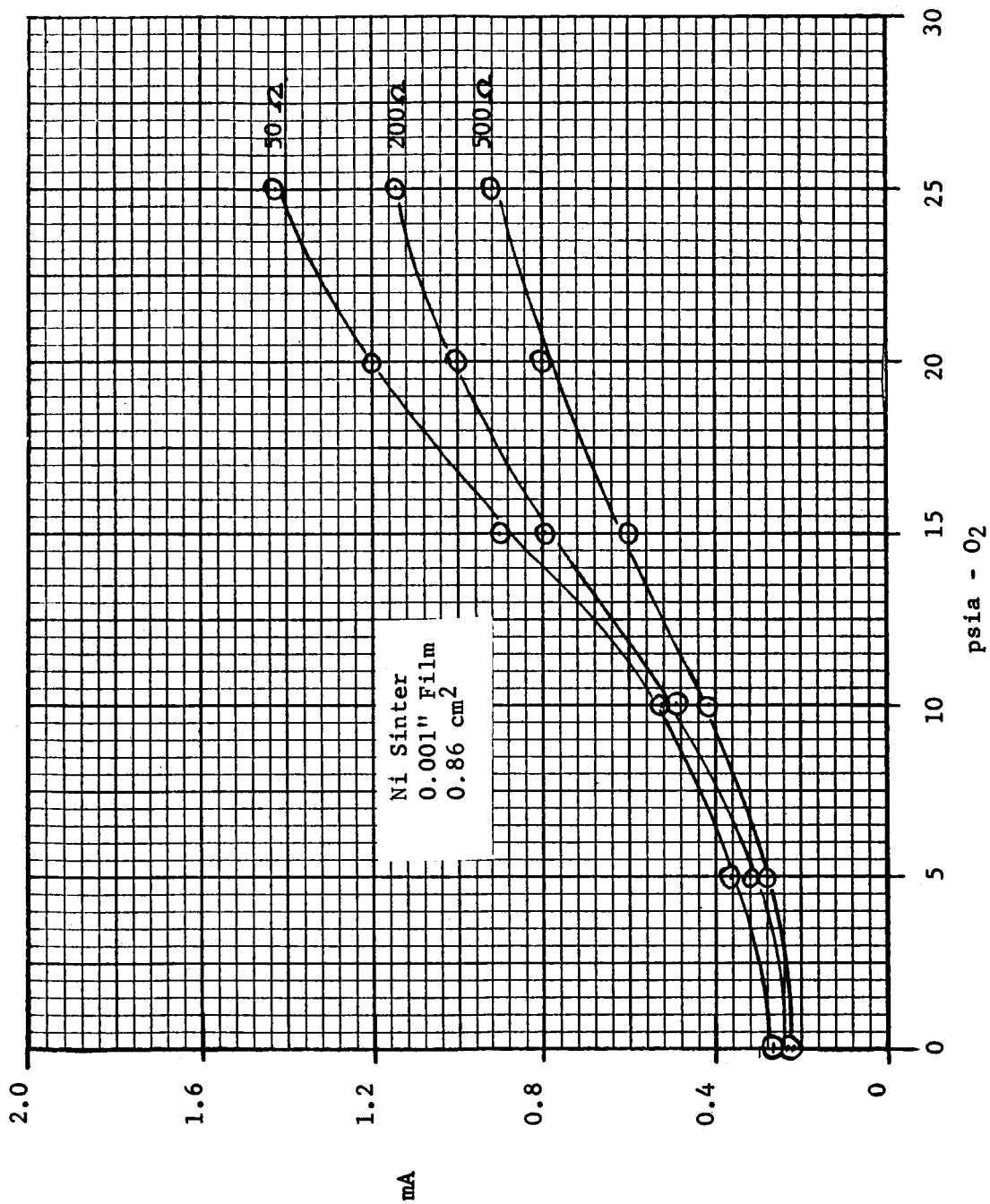


Figure 6: Electrode Response - V

TABLE II
SIGNAL ELECTRODE EVALUATION AT 25°C

Electrode Material	Film Thickness(1)	0.86 cm ² Electrode(2)	9.8 cm ² Electrode(2)
Ni Sinter	0.001"	B	A
Ni Sinter	0.0005"	B	B
Ni Sinter	0.00025"	C	C
Ni Mesh	0.001"	B	B
Ni Mesh	0.0005"	B	B
Ni Mesh	0.00025"	C	C
Au Mesh	0.001"	D	D
Au Mesh	0.0005"	B	B
Au Mesh	0.00025"	B	D
Pt Coated Ta Mesh	0.001"	B	B
Pt Coated Ta Screen	0.001"	B	B

(1) Skived Dilectrix Teflon Sheet, sintered on to one side of electrode.

(2) Rating is as follows:

- A. Non-linear, not suitable for indicator use.
- B. Non-linear, curves upward, suitable for indicator use.
- C. Sigmoid curves, not suitable for indicator use over wide pressure range.
- D. Erratic behavior on load.

TABLE III
SUMMARY OF SIGNAL ELECTRODE POLARIZATION TESTS

ELECTRODE				TEST		
No.	Size	Material of Substrate	Film in	Temp °C	Res. Range Ohms	Remarks(2)
1	9.8	Nickel Sinter	0.001"	-10 0 10 25 35	10-200 DO DO DO DO	Linear to 25 PSIA(4) Linear Linear Curves Upward(3) Linear
1	0.86	Nickel Sinter	0.001"	-10 0 10 25 35	20-200 DO DO DO DO	Linear Linear Curves Upward(3) Curves Upward(3) Linear to 25 PSIA(4)
2	9.8	Nickel Sinter	0.0005"	-10 0 10 25 35	10-200 DO DO DO DO	Curves Upward(3) Curves Upward(3) Linear Curves Upward(3) Curves Upward(3)
2	0.86	Nickel Sinter	0.0005"	-10 0 10 25 35	20-200 DO DO DO DO	Curves Upward(3) Curves Upward(3) Linear Linear Linear
4	9.8	Nickel Mesh	0.001"	10 25 35	10-200 DO DO	Test Cell Failed(6) Linear Linear
4	0.86	Nickel Mesh	0.001"	25 35	20-200 DO	Linear Linear

NOTES TO TABLE III

1. Refer to Table I
2. Curves plotted mA-signal electrode to cadmium electrode vs. PSIA oxygen 0-30.
3. Curve increases in slope with increase in oxygen pressure.
4. Curve flattens at higher oxygen pressure.
5. Curve linear 0-20 PSIA, then curves upward.
6. Nickel-cadmium part of test cell shorted, further tests abandoned.

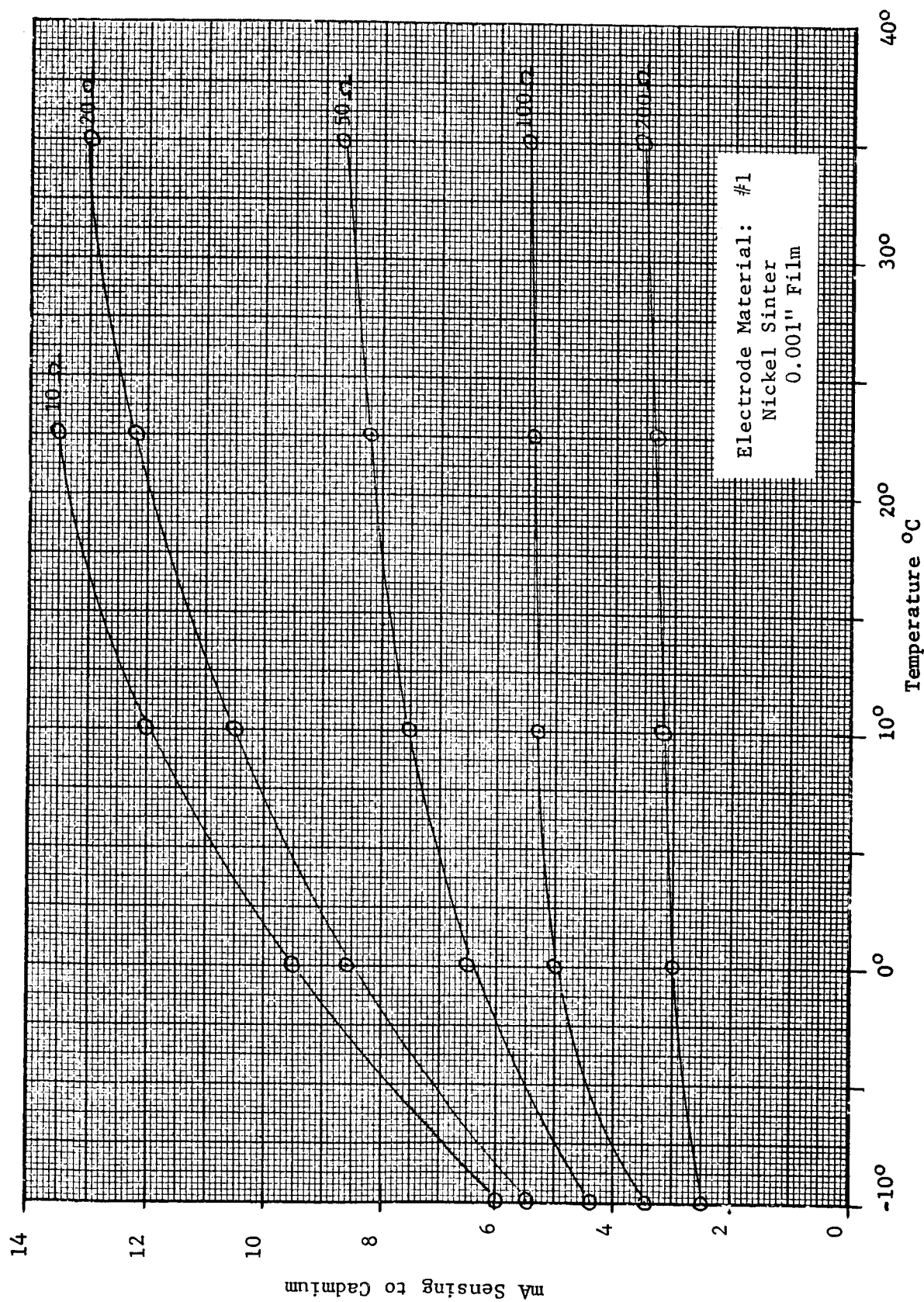


Figure 7: Effect of Temperature on Electrode Response at 20 PSIA

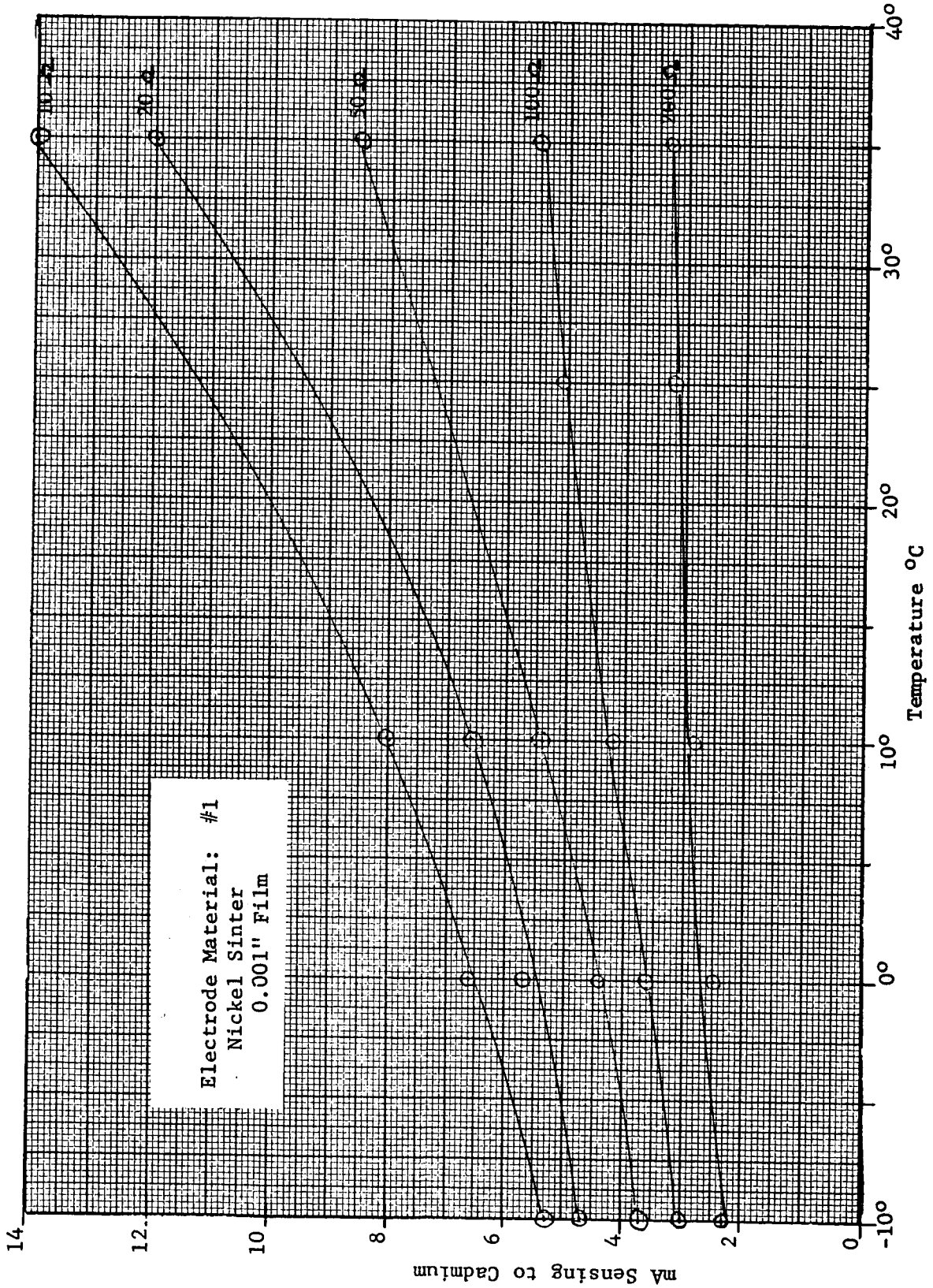


Figure 8: Effect of Temperature on Electrode Response at 30 PSIA

2.1.3 Analysis of Results

In the cell the signal electrode is coupled with a cadmium electrode. Oxygen reacts at the signal electrode to form hydroxyl ions which serve to complete the cell reaction.



The rate of oxygen reduction is a function of the electrode material and tends to be proportional to either the area of the electrode, the internal cell impedance, the external impedance, or to the rate of diffusion of oxygen to the electrode surface. Any one of the factors may be controlling; thus, the current obtained from a small area electrode is usually less than that from a larger area electrode and, in fact, will be proportional to the electrode area if all the other factors are non-limiting. Since the desire is to produce an electrode that gives a current proportional to the pressure of oxygen, it is necessary that the electrode current be controlled by a pressure sensitive mechanism such as the rate of diffusion of oxygen to the electrode.

The diffusion rate of oxygen through a film into a closed volume is given by:

$$r = k (P_1 - P_2) A/d$$

where r is the rate of diffusion

k is a constant for a given film and temperature

P_1 is the external pressure

P_2 is the internal pressure

A is the film area

d is the film thickness

The linear response of an electrode arises from its effect on P_2 . The electrode is under diffusion control when all of the oxygen arriving at the electrode is reacted immediately and P_2 is reduced to virtually zero. Therefore, the diffusion rate and, hence, the electrode current is proportional to P_1 .

If the internal plus external resistance is too large, the electrode may show a linear but very flat response. This is shown in Figure 4. With an external resistance of 500 ohms, the curve is linear but has a small slope. With a decrease to 200 ohms, a much steeper slope is obtained. If the internal resistance is too high, the response of the electrode may "saturate" at some pressure and give a flat response for all higher pressures. This is shown in Figure 2. If the electrode area is large, the response may be flat since the load resistance cannot be reduced to bring the electrode into the diffusion controlled state. This is also shown in Figure 2.

The simple relation between pressure and electrode response holds only to the first approximation, and it is not surprising that non-linearities can and do occur at low load resistance and higher gas pressures. As long as the non-linearity is positive, (i.e., the response increases faster than the pressure) the response may be useful for signal purposes.

2.2 Task II - Oxygen Recombination Electrode

2.2.1 Experimental Details

The test cells described in the previous quarterly report were modified in order to facilitate the removal of heat generated during cell operation. The fact that the heat generated may significantly effect the results is illustrated in Figure 9. In both cases, the electrodes were assembled in the cells illustrated in the previous quarterly report, and were insulated from the can wall by plexiglass face plates and the oxygen atmosphere.

Curve A is for a cell being operated in an ambient temperature of -20°C , while curve B was obtained in an ambient of 0°C . Both electrodes appear to have improved performance (i.e., lower polarization) at the higher current densities. This is, of course, unreasonable, and so the decreased polarization must have been the result of an increase in the temperature of the working electrodes.

In order to minimize this heating, it was necessary to provide good thermal contact between the working electrodes and can. This was accomplished by welding the sintered nickel counter electrode directly to the can wall and assembling the cell as shown in Figure 10.

The modified cells also contain thermocouples, making it possible to measure the temperature of the test electrode during operation. Electrode temperatures measured to date in the modified cells are only 2 to 3°C above the nominal ambient. This modest temperature rise is insufficient to result in the effects observed in the previous cells. All other details are as outlined in the previous quarterly report.

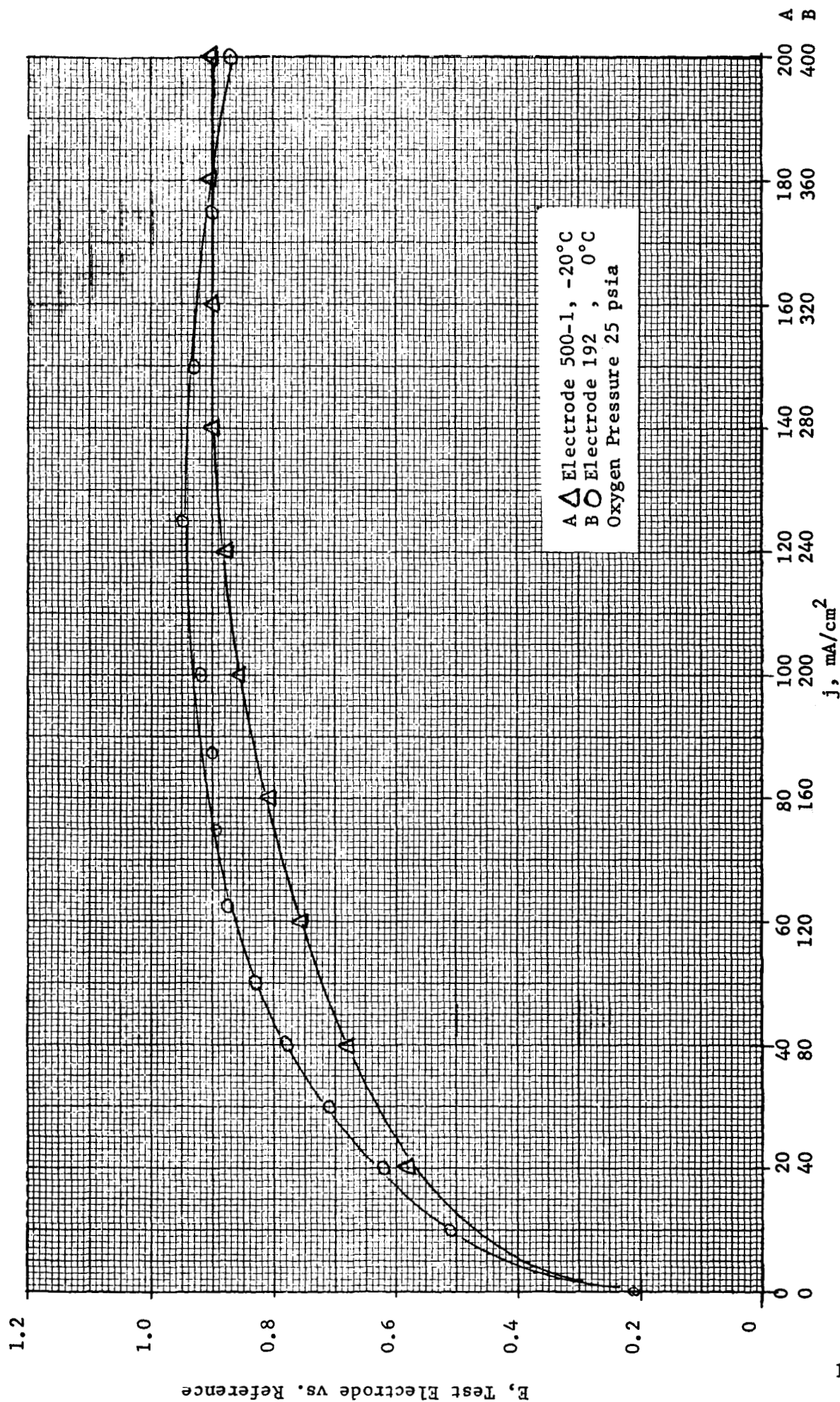


Figure 9: Polarization Curves Showing Effect of Internal Heating

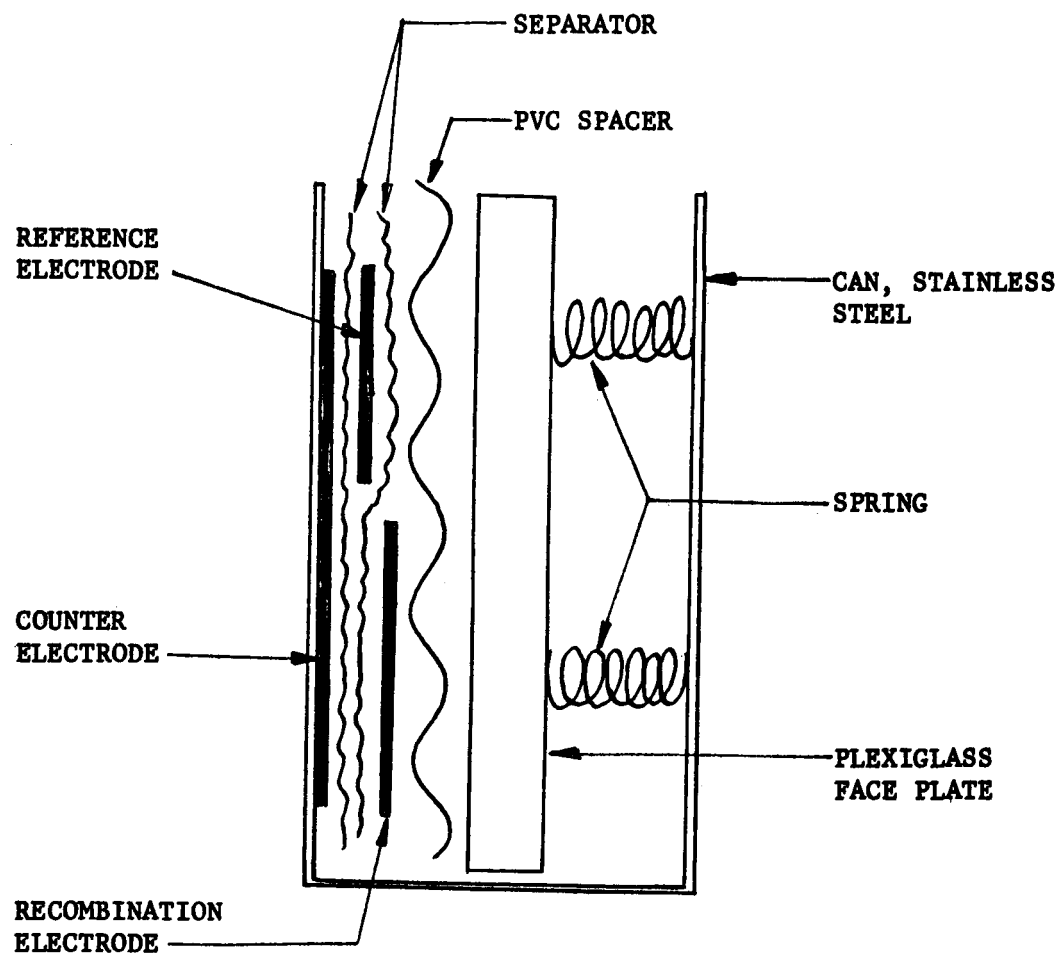


Figure 10
Recombination Electrode Test Cell Assembly

2.2.2 Evaluation of Fuel Cell Electrodes

Polarization curves were obtained on a variety of fuel cell electrodes in order to determine their suitability as recombination electrodes. Data were obtained at 25°C and -20°C, using the test cells described above.

The electrodes evaluated included General Electric Research and Development Center Type 511 and American Cyanamid Company Type E and LBB-3CG. Polarization curves are presented in Figures 11 and 12. As can be seen, the 25°C performance of all electrodes evaluated are comparable. At the low temperature, however, the improved electrodes developed in this program offer the best performance.

2.2.3 Electrode Reproducibility and Stability

Two series of tests were performed in order to determine whether our electrode fabrication process would enable us to produce electrodes of consistent quality.

The first test series was performed using three electrodes from the same lot, prepared during the second week in October. Polarization curves illustrating the range in performance observed at both 25°C and -20°C are shown in Figure 13.

The second series compared the performance of electrodes prepared in December with those prepared in October. Again the results were comparable, demonstrating that electrodes of consistent quality can be prepared using our present techniques.

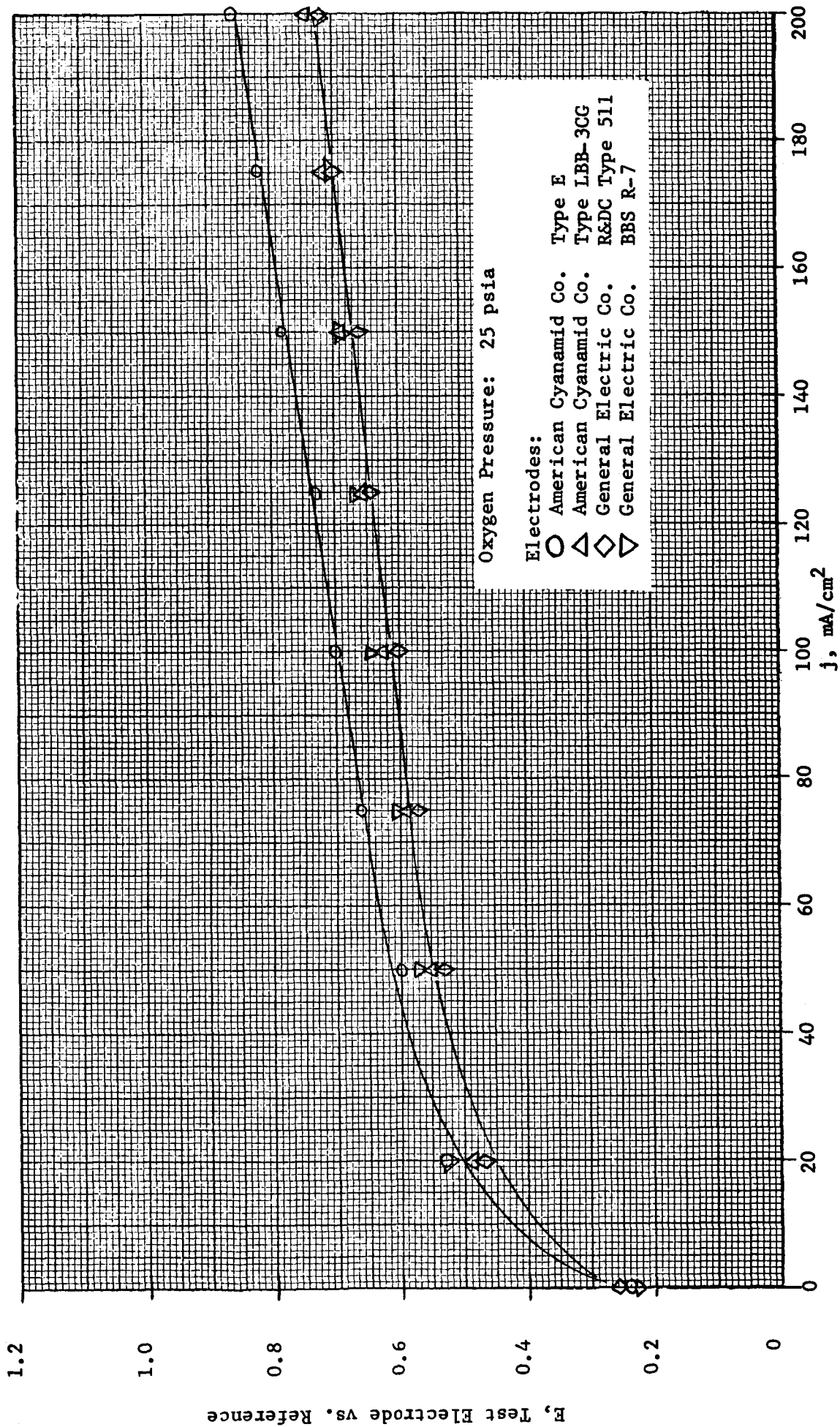


Figure 11: Performance of Various Fuel Cell Electrodes at 25°C

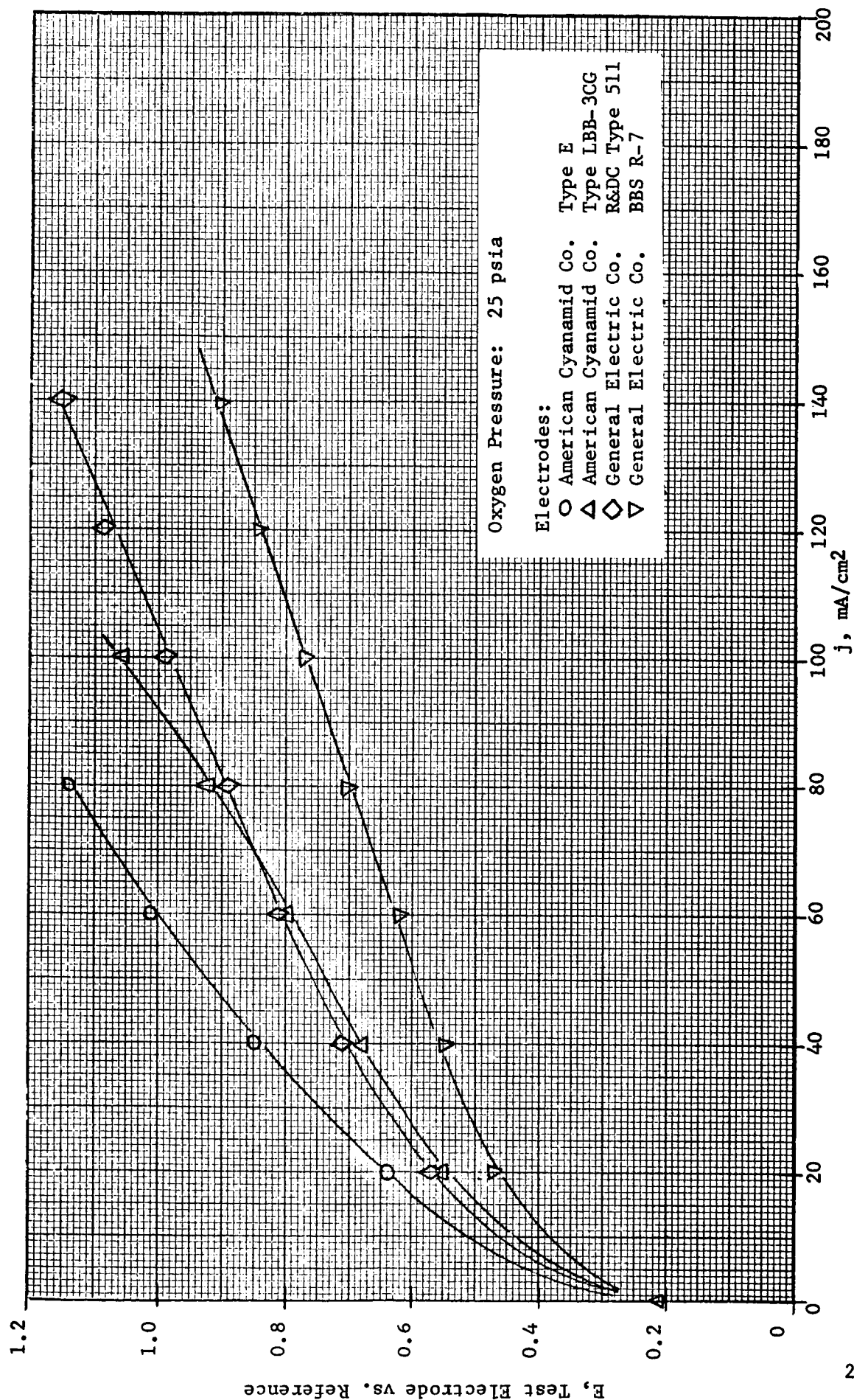


Figure 12: Performance of Various Fuel Cell Electrodes at -20°C

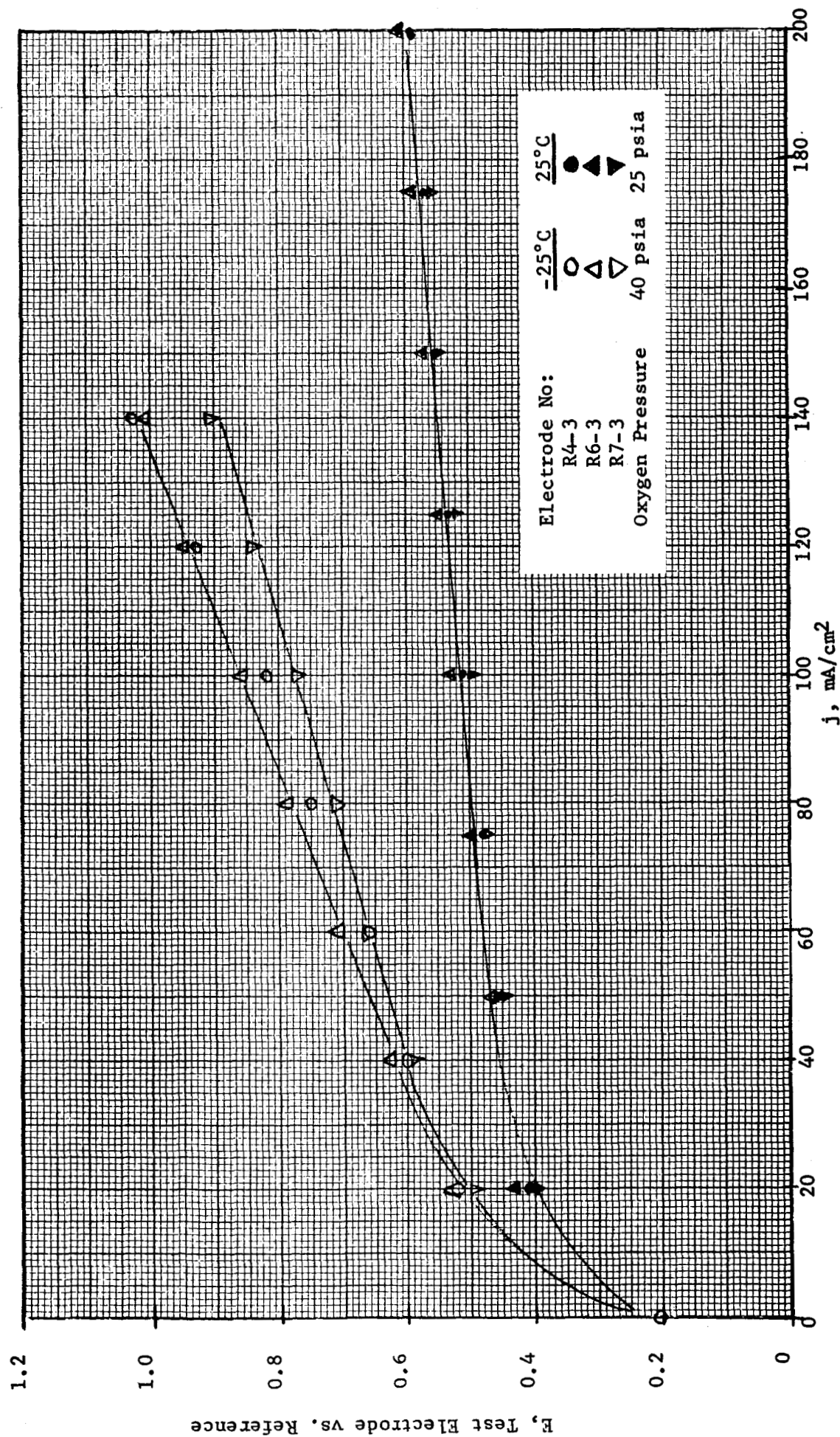


Figure 13: Reproducibility of Improved Recombination Electrodes

An investigation of the stability of recombination electrodes has been performed. The program consisted of determining the initial performance of the electrode at both 25°C and -20°C with 25 PSIA oxygen pressure. The electrodes were then subjected to storage at 40°C under 25 PSIA oxygen and the -20°C polarization checked at regular intervals. The high temperature storage should accelerate any degradation process, and the extent to which these processes affect electrode performance should readily be seen at -20°C.

The results of one such test are shown in Figure 14. In this test the electrode was subjected to 64 hours of storage at 40°C. The slight shift in the polarization curve is not due to a change in the test electrode, but rather a change in the reference electrode as a result of the high temperature exposure. The reference electrodes used in these cells is a piece of partially charged positive plate, and it is reasonable to expect their state-of-charge, and hence their potential, would be affected by such a high temperature storage.

In order to demonstrate this, a "Normalized Polarization" may be computed by subtracting the open circuit voltage from the voltage observed at each current. Such normalized polarization curves should be identical if, indeed, the shift is due to the reference electrode. Such normalized curves for the above experiment are presented in Figure 15 and the curves do superimpose.

An additional test of electrode stability was performed with a total storage time of 144 hours. The test cells were removed from the high temperature periodically and their -20° polarizations were checked. Results of this test are shown in Figure 16. Again, the curves superimpose, indicating that the high temperature storage does not effect electrode performance.

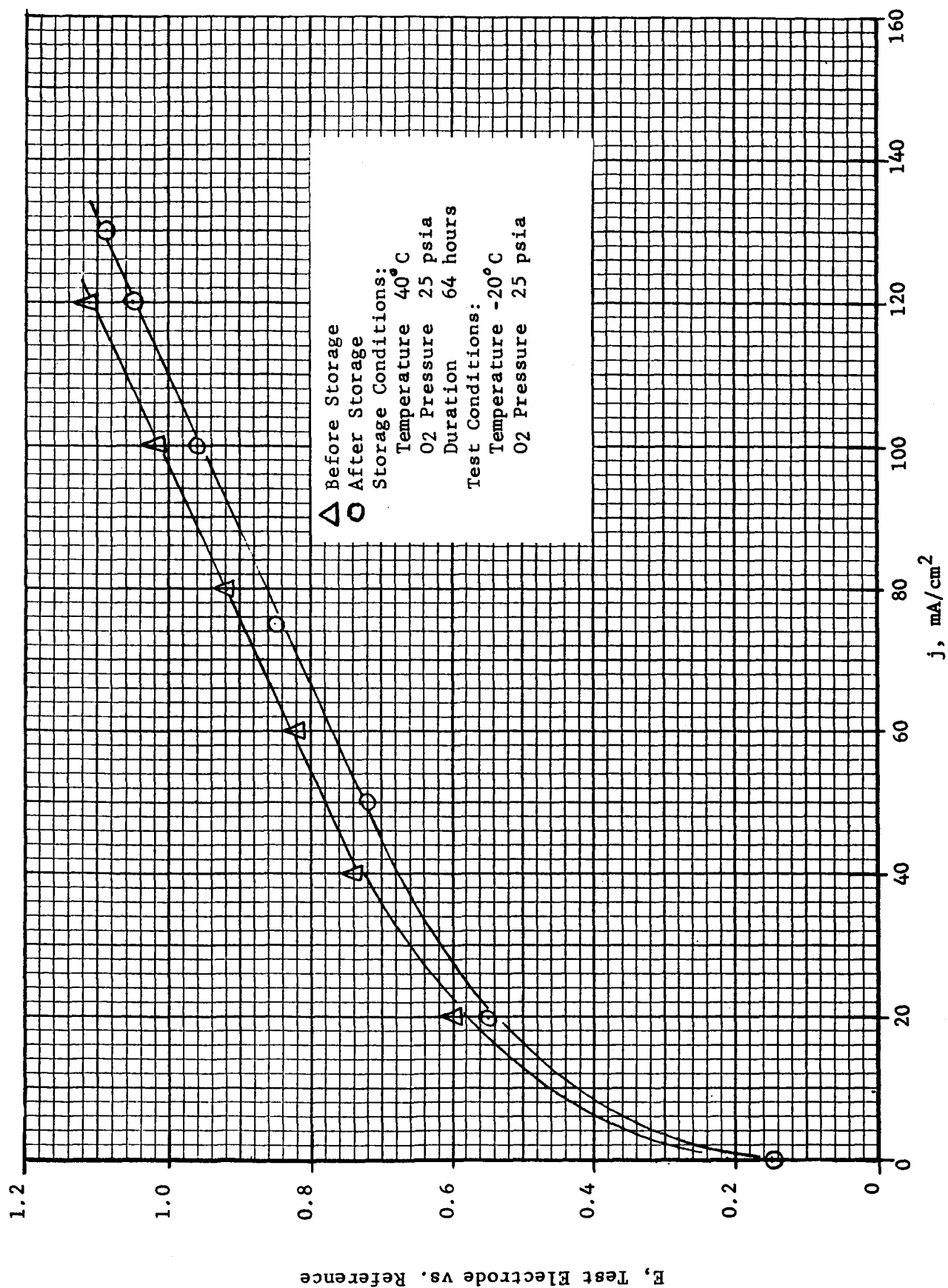


Figure 14: Effect of Storage on Electrode Polarization

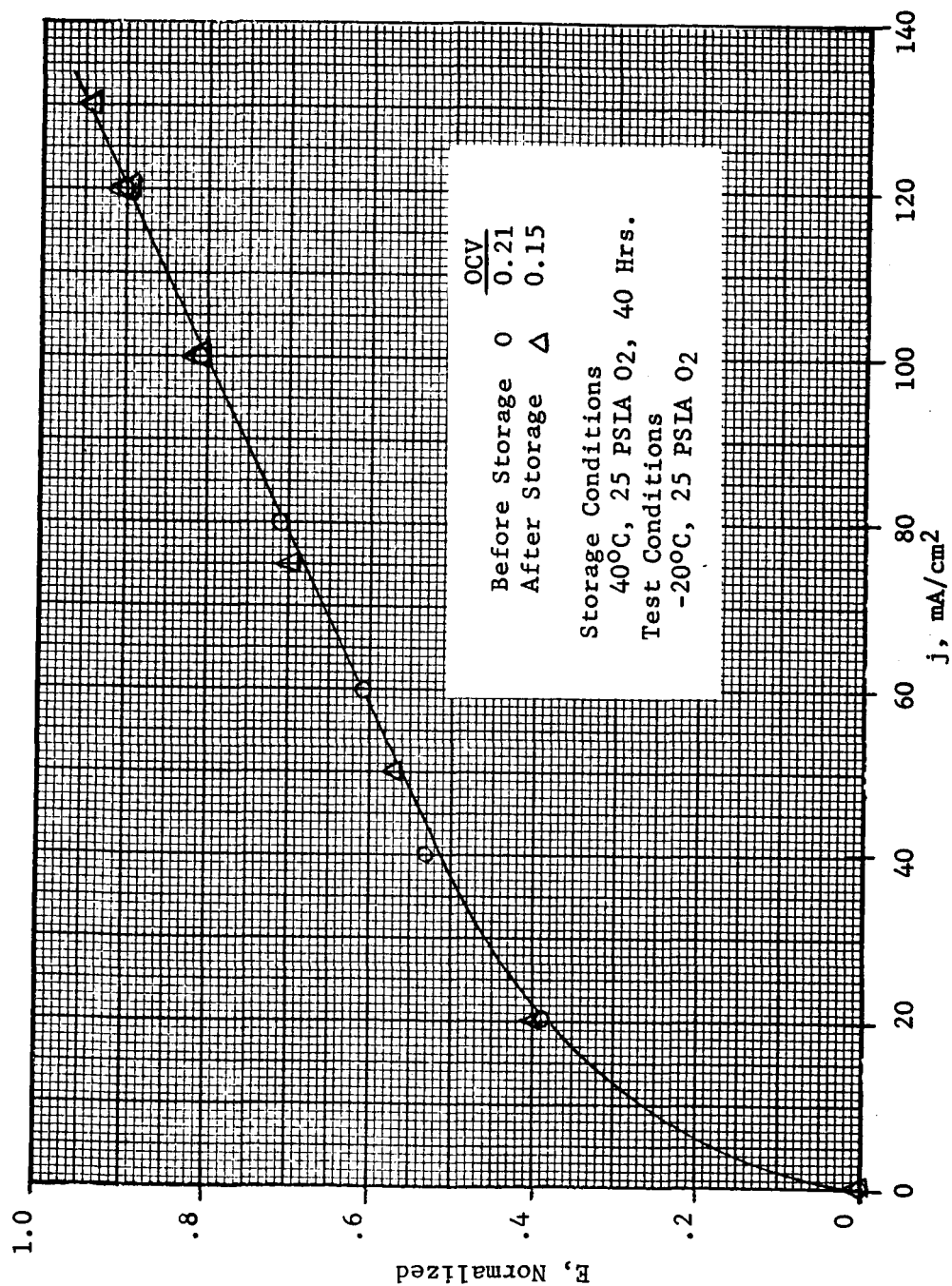


Figure 15: Normalized Polarization Curves for Electrode Before and After 400C Storage - I

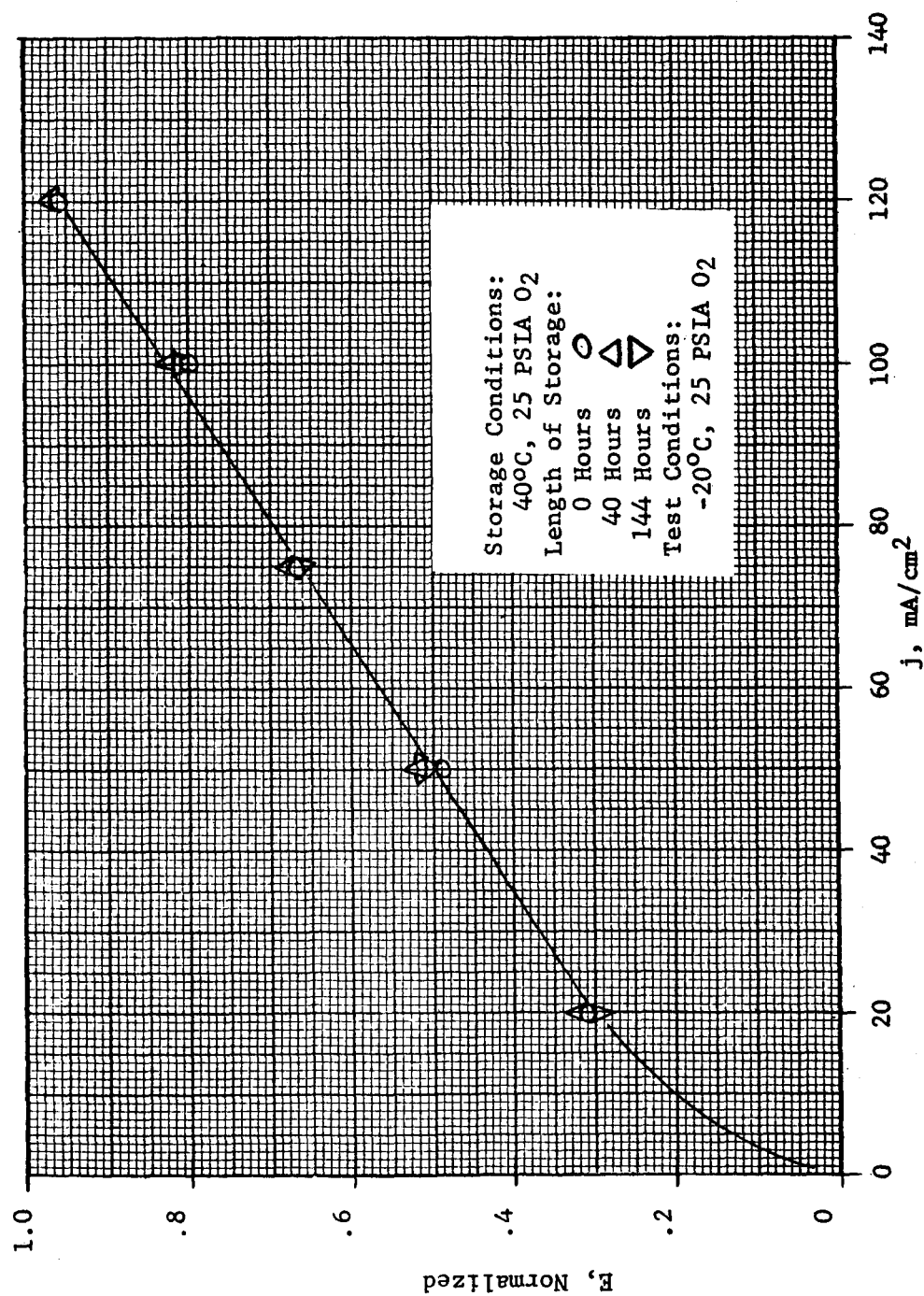


Figure 16: Normalized Polarization Curves for Electrode Before and After 40°C Storage - II

2.3 Task III - Negative Plate Evaluation

2.3.1 Experimental Details

Three lots of negative plate were received from manufacturing for evaluation of ampere-hour stability and oxygen recombination ability. A sufficient number of plates to construct 12 cells from each lot were cut to size and subjected to standard aerospace formation cycles. The positive plates for all cells were taken from one lot, and were formed along with the negatives. The plates were then washed free of electrolyte and dried.

Plate packs of nominal 4AH configuration were assembled, using standard non-woven nylon separator. Sufficient 34% KOH electrolyte was added to produce semi-starved cells. The cells were fitted with pressure gauges and sealed with epoxy.

In order to determine the ampere-hour stability of the plate, a regime known to promote negative plate fading was employed. The regime was as follows:

Temperature:	Room ambient
Charge:	400 mA for 16 hours
Discharge:	4.0 A to 1.0 V
Rest:	7 hours
5 cycles per week	

Cycling was continued until the discharge capacities of the cells stabilized. This required 16 to 18 cycles.

The oxygen recombination ability of the negative plate is determined by observing the steady state pressures in a cell at various rates of continuous overcharge. The overcharge rates employed were 400 mA (C/10), 500 mA (C/8), and 800 mA (C/5). The C/5 overcharge rate is the upper threshold

for continuous overcharging of sealed cells without recombination electrodes and was employed in this test in order to increase the amount of data available for use in selecting a suitable plate lot.

2.3.2 Results and Discussion

The capacity versus cycle data for the three lots evaluated are present in Figure 17. As can be seen, two lots, 6939 and 6979 behaved essentially the same. The third lot, 8104, faded to a greater extent.

In addition to the cycling regime, the extent to which negative plates fade is related to the state-of-charge of the plate at the time of cell assembly, the higher the state-of-charge the less fading. The difference, therefore, between lot 8104 and the others may be due to a difference in the original state-of-charge of the plates.

The pressures developed in the cells during the continuous overcharge tests were in the ranges normally observed when charging at the rates employed. The pressures developed in the lot 6979 cells were, however, somewhat lower than the others.

Negative plate from lot 6979 exhibited the best balance of properties - low fading and low continuous overcharge pressures - and has been selected for use in prototype and final cells.

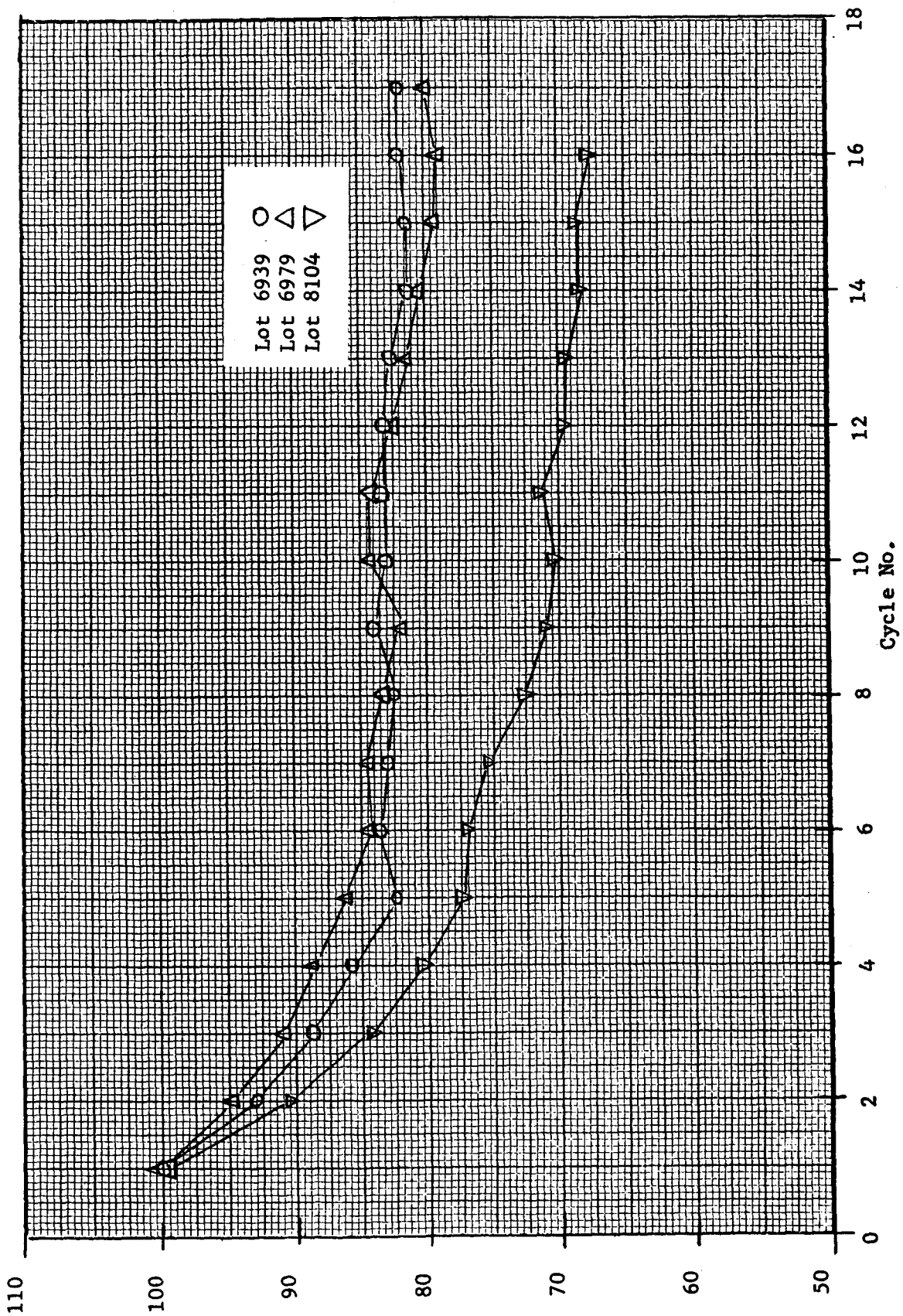


Figure 17: Ampere - Hour Stability of Negative Plates

3.0 WORK PLANNED FOR NEXT QUARTER

3.1 Task I - Oxygen Sensing Electrode

Phases to be completed during the next quarter include:

1. Completion of temperature runs.
2. Construction and test of signal electrode cells on a cyclic basis at various temperatures. In these tests, the sensing electrode will cut off charge and discharge of oxygen generation; the pressure will be monitored.
3. Design and initial construction of test cells with both auxiliary electrodes.

3.2 Task II - Oxygen Recombination Electrode

Prototype cells incorporating these electrodes will be cycled in order to determine the capability of these electrodes to maintain low oxygen pressures up to the end of charge as a function of circuit parameters, location within the cell, and charging conditions. Cycling will be conducted at three temperatures, -20°C, 25°C, and 40°C, and three depths of discharge, 25, 50, and 75%.

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